

UNCLASSIFIED

AD 296 889

*Reproduced
by the*

**ARMED SERVICES TECHNICAL INFORMATION AGENCY
ARLINGTON HALL STATION
ARLINGTON 12, VIRGINIA**



UNCLASSIFIED

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

63-2-4

296 889

AZUSA PLANT

STRUCTURAL MATERIALS DIVISION

STRESS-CORROSION CRACKING
OF HIGH-STRENGTH ALLOYS

Contract DA-04-495-ORD-3069

A Report To

U.S. ARMY ORDNANCE CORPS
FRANKFORD ARSENAL

Report No. 0414-01-8 (Quarterly)

February 1963

12

CATALOGED BY ASTIA

29 6889



AEROJET-GENERAL CORPORATION

AZUSA, CALIFORNIA

This is the eighth in a series of quarterly progress reports submitted in partial fulfillment of the contract. It constitutes the second quarterly progress report for the one-year continuation of the original two year program.

This report covers the period 1 October through 31 December 1962. It was written by R. B. Setterlund, who was supervised by A. Rubin.

Approved by:



F. L. Jordan, Head
Metallics and Refractories Department
Structural Materials Division

CONTENTS

| | <u>Page</u> |
|--|-------------|
| I. OBJECTIVES _____ | 1 |
| II. SUMMARY _____ | 1 |
| III. WORK PROGRESS _____ | 2 |
| A. Introduction _____ | 2 |
| B. Test Procedures _____ | 3 |
| C. Program Status _____ | 4 |
| D. Test Results To Date _____ | 5 |
| IV. FUTURE WORK _____ | 8 |
| A. Master Program _____ | 8 |
| B. Further Experiments _____ | 8 |
| C. Metallography and Electron Microscopy _____ | 9 |

Table

| | |
|--|---|
| Master Schedule, Third-Year Program _____ | 1 |
| Chemical Analysis and Mechanical Properties of Maraging Steels _____ | 2 |
| Chemical Analysis and Mechanical Properties of 6Al-4V Titanium _____ | 3 |
| Stress Corrosion of 6Al-4V Titanium in Various Environments _____ | 4 |
| Stress Corrosion of 20%-Nickel Maraging Steel in Various Environments _____ | 5 |
| Stress Corrosion of 18%-Nickel Maraging Steel in Various Environments _____ | 6 |
| Evaluation of Coatings on H-11 Steel for Preventing Stress Corrosion Cracking _____ | 7 |

Figure

| | |
|--|---|
| Insulated Phelps Bent-Beam Specimens _____ | 1 |
| U-Bend Test Specimens _____ | 2 |

CONTENTS (cont.)

| | <u>Figure</u> |
|--|---------------|
| Elox-Notched Specimen _____ | 3 |
| Stress-Corrosion Test Setup for Center-Notch Specimens _____ | 4 |
| Stress-Corrosion Crack Pattern on 20%-Nickel Maraging Steel _____ | 5 |
| Crack Propagation Study on 20%-Nickel Maraging Steel in Salt Water _____ | 6 |
| Stress-Corrosion Crack Pattern on 18%-Nickel Maraging Steel _____ | 7 |
| Photomicrographs of Stress-Corrosion Cracking in 18%-Nickel Maraging Steel _____ | 8 |

I. OBJECTIVES

The objectives of the program extension are outlined below:

- A. Investigation of the stress-corrosion cracking characteristics of at least three new high strength alloys of interest for rocket motor case applications. These alloys will be 6Al-4V titanium, 18%-nickel maraging steel, and 20%-nickel maraging steel, in addition to limited testing of vacuum-melted 9Ni - 4Co steel.
- B. Study of the environmental parameters that could affect the rate and extent of stress-corrosion cracking.
- C. Determination of the effect of material parameters (composition, strength level, welding, and microstructure) on stress-corrosion susceptibility.
- D. Continuation of the evaluation of protective coatings and other techniques for preventing stress-corrosion cracking.

II. SUMMARY

Data to date show that the 6Al-4V titanium alloy is immune to stress-corrosion cracking under the test conditions of this program in both the annealed and in the quenched-and-aged conditions. It has also been found that stress-corrosion cracking occurs in both 18%- and 20%-nickel maraging steels, after short exposure to specific environments. The 20%-nickel variety, if not cold-worked, is found to crack in a branching intergranular pattern when exposed to distilled water, salt water, or a high-humidity atmosphere. However, when this same alloy is cold-worked before aging, it appears to become immune to cracking in all three of these environments. The 18%-nickel maraging steel has, to date, been tested only in the cold-worked-and-aged condition. In

II Summary (cont.)

this condition, the alloy cracks in distilled water and in a high-humidity environment; the cracking pattern suggests possible failure along slip-planes.

III. WORK PROGRESS

A. INTRODUCTION

Since the initiation of the original test program two years ago, to investigate the stress-corrosion cracking characteristics of high-strength alloys, a number of new high-strength steels have been receiving increased attention for use in constructing rocket motor cases. The third year test program is directed to the study of three of these new alloys as well as of one titanium alloy presently being used for the same application.

The test environments, substantially the same as those evaluated in the original two-year investigation, are as follows: (1) distilled water; (2) tap water; (3) salt water; (4) sodium-dichromate-inhibited water; (5) soluble oil-inhibited water; (6) air; (7) high humidity atmosphere; (8) tri-chloroethylene; (9) cosmoline; and (10) solid propellant. These are considered to be environments representative of those to which rocket motor cases would normally be exposed during fabrication, processing, or storage. One additional environment will be included in the new program, that of sea coast exposure.

The test methods being used in this investigation employ bent-beam, U-bend, and center-notch specimens. Evaluation of results includes micro-structural studies, using both standard metallographic and electron microscopic techniques, to attempt to associate the failure mechanism with specific micro-structural characteristics of the materials.

An evaluation of protective coatings and surface treatments to prevent stress-corrosion cracking is also being conducted.

III Work Progress (cont.)

B. TEST PROCEDURES

1. Bent-Beam Tests

The bent-beam test is the primary test method used in this program. Figure 1 shows an insulated bent-beam fixture with test samples mounted. Polycarbonate blocks $7.000 \pm .001$ in. apart, attached to a stainless steel holder, support the test specimen and insulate it from the holder. Specimens are cut to exact length to give a maximum calculated outer fiber stress of 75% of the 0.2% offset yield strength. A four-point loading device is being used to place test specimens into the holders. By using four-point loading in this pre-stressing device, possible local plastic deformation, which may have occurred during some earlier tests with a three-point loading device, is now eliminated. Samples which have been stressed in this manner and then released show no apparent distortion.

2. U-Bend Test

Figure 2 shows a U-bent test sample. This test is being used to accelerate failure times in environments where the center-notch test, described below, cannot be used.

3. Center-Notch Test

Figure 3 shows the test specimen configuration used in the accelerated center-notch test. It consists of a $1\frac{3}{4}$ - by 8-in. tensile specimen containing a central notch. This notch is produced by a two-step process. First, a 0.06- by 0.05-in. slot is Elox-machined and extended at each end by very narrow Elox-machined notches of 0.001-in. root radii. Second, an extension of these notches is produced by fatigue cycling to obtain fatigue cracks of controlled dimensions.

These center-notch specimens are tested in Baldwin creep-test machines, one of which is shown in Figure 4. Dead-weight loading is applied to a 20:1 lever arm to stress the specimen to 75% of its notched tensile strength.

III Work Progress, B (cont.)

The test solution is poured into a polyethylene cup cemented to the specimen in the notch area before the load is applied. An electric timer records the elapsed time at fracture.

C. PROGRAM STATUS

The titanium alloy, 6Al-4V has been under test for several weeks in two of the three conditions scheduled in Table 1. Welded plates have now been fabricated and X-rayed for joint integrity, and specimens are being prepared for testing of the remaining condition, welded joints.

The 20%-nickel maraging steel is under test in the annealed-and-aged condition as well as in the 75% cold-worked condition. The 50% cold-worked material was reannealed, by mistake, at the mill after cold-working; it was therefore necessary to prepare a new heat. Delivery of this material is scheduled for mid-February.

The 18%-nickel maraging steel is under test in the 50% cold-worked condition. Sufficient 50% cold-worked material was obtained so that part of the heat was reannealed and aged for the testing of condition 1-1 (Table 1). This will serve as a substitute for the annealed material on order which has not yet been received. The determination of the effect of titanium content of this alloy on its stress-corrosion cracking characteristics (another objective of this program) is being conducted with limited quantities of three additional heats obtained from another program. The chemical analysis of these heats (heats 477, 448, and 476, shown in Table 2) shows a titanium content varying from 0.40 to 1.00%.

The 9Ni-4Co vacuum cast alloy is now scheduled for delivery at the end of February. Shipment delays were caused by difficulties at the mill in producing a satisfactory heat.

III Work Program (cont.)

D. TEST RESULTS TO DATE

1. 6Al-4V Titanium

Testing is well along on the 6Al-4V titanium alloy. The chemical analysis and mechanical properties of the material are shown in Table 3. This alloy is being tested in the three most common metallurgical conditions - annealed, quenched-and-aged, and welded. All titanium samples are given a post-heat-treat machining operation. It was found in previous work with this alloy that removal of 0.010 in. of material from each surface improves ductility, and the material is so-processed in motor case fabrication. Results of testing to date are shown in Table 4. No failures have occurred in the annealed or heat-treated conditions in any of the tests. Titanium sheets have been welded at Aerojet, the weldments X-rayed to verify their soundness, and specimens fabricated.

2. 20-Nickel Maraging Steel

The chemical and mechanical properties of the 20%-nickel maraging steel are shown in Table 2. These data show that a -100°F refrigeration treatment prior to aging improved the properties of the annealed material. Environmental test results to date are shown in Table 5. The annealed-and-aged material was found to fail rapidly in both distilled water and in the 3% NaCl solution. The material also failed in the high humidity atmosphere but required a slightly longer exposure time.

Examination of the cracked specimens shows a branching crack pattern, as illustrated in Figure 5. The same figure also shows a photomicrograph of a crack which indicates cracking to be intergranular in nature.

This crack pattern, shown in Figure 6, was found to be quite slow in developing. Figure 6 drawings are based on sketches made while the crack was forming. For this particular sample, it was found that crack propagation required a longer time than did crack initiation. For this material,

III Work Program, D (cont.)

the center-notch tests showed roughly the same failure times in salt water and distilled water as the bent-beam tests. One bent-beam failure occurred in the dichromate solution, while two other samples in the same solution have sustained, to date, 600 hours without fracture. No failures have occurred during the center-notch testing in dichromate solution.

Two interesting results have been noted in the testing of the 20%-nickel alloy. One is the high resistance to stress-corrosion cracking of the cold-worked-and-aged material in environments where the annealed-and-aged material with lower mechanical properties cracks quickly. Another is the complete absence of cracking of either alloy, to date, in the tap water environment.

3. 18-Nickel Maraging Steel

The chemical and mechanical properties of the 18%-nickel maraging steel are shown in Table 2. This heat was originally intended to be tested in the cold-worked-and-aged condition only; however, because of its relatively low titanium content we propose to test it in the annealed-and-aged condition also. Results of tests to date are shown in Table 6. Cracking in this alloy occurred most rapidly in the high humidity environment. The mode of crack propagation, however, appeared quite different from crack patterns previously encountered.

Figure 7 shows the cracking pattern on the surface of a cold-worked-and-aged, 18%-nickel, bent-beam specimen after 10 days in water-saturated air, at 140°F. It depicts the multiple cracks which developed across the width of the specimen. Curiously, the direction of propagation down through the specimen thickness (shown in the bottom panel of Figure 7) is at a sharp angle from the perpendicular, forming a cross-hatch pattern; such a crack pattern is sometimes indicative of a slip-plane mode of cracking, although the information at present is insufficient to definitely establish this fact. Figure 8, a photograph, made at greater magnification, of some of these cracks indicates initiation of branching cracks developing perpendicular to the main fracture lines.

III Work Program, D (cont.)

The nature of these cracks will be further investigated to determine whether the fractures actually occur along Widmanstätten lines (indicating heat-treat or cold-working dependency) or along slip planes (indicating intercrystalline structural weakness). Residual stress measurements will be performed by X-ray diffraction methods to determine the critical stress level, and optical and electron fractography studies will be performed on the fracture surfaces to indicate the microstructural changes associated with the cracking process. A detailed examination of the cracking pattern will also be made to establish whether cracking is intergranular or transgranular in nature.

4. Coatings Evaluation

The testing of various coatings designed to prevent stress-corrosion cracking in continuing (Table 7). The coatings are in addition to the three tested during the first two-year program. In the present tests, H-11 steel is being used as the base material; the test environments are 3% salt water, high humidity atmosphere, and sea coast exposure. H-11 steel was selected as the base material for the coating evaluation because of its characteristic short failure time, when exposed unprotected to these environments.

Presently, five coatings are under test. The 80%-aluminum, epoxy-base coating represents the principle of anodic protection by employing a metal coating that is galvanically anodic to the base material. This coating, however, appears to be unsuccessful in that short failure times are being obtained. A 70%-titanium, epoxy-base coating has been similarly unsuccessful. The zinc silicate coating under test suffered from poor adhesion and consequently afforded little protection. The 463-4-8 epoxy coating successfully protected the base metal for 500 hours of immersion in 3% salt water. (This can be compared to failure times of approximately 2 hours in earlier tests with uncoated base metal.) After this period, the coating separated from the base material, and stress-corrosion failure of the metal then followed. Epoxy coating 463-1-5, although originally developed as a primer, has been used very effectively in

III Work Program, D (cont.)

the aircraft industry, when applied as a single coating for salt water resistance. It appears to be the most promising coating in the present program, on the basis of preliminary results which show no failures in salt water after 700 hours of exposure.

IV. FUTURE WORK

A. MASTER PROGRAM

Work will be continued to fulfill the schedule shown in Table 1. Sheets of the 6Al-4V titanium alloy have been welded and specimens representing code group G-W in Table 1 will soon be in test. The annealed-and-aged 18%-nickel maraging steel (group I-1) is in the final stage of machining and specimens will be tested in January. The exposure to solid propellant and the marine atmosphere testing will be started when samples are prepared.

B. FURTHER EXPERIMENTS

A limited amount of additional material, consisting of three different heats of 18%-nickel maraging steel, as listed in Table 3, has been obtained from another program. This material will be used for bent-beam and center-notch specimens of each heat, in the annealed-and-aged and cold-worked-and-aged conditions, to determine the effect of titanium content of the alloy on stress-corrosion cracking. Because of the limited quantity of material available, these tests will be run in salt water and high humidity environments only.

It has been noted that to date no specimens have failed in the tap water environment. This condition could be the result of either the higher pH of the tap water (7.8), as opposed to distilled water (7.0), or to the inhibitory effect of dissolved salts in the tap water. To investigate this condition, center-notched samples of 20%-nickel maraging steel will be tested in distilled water of various pH levels.

IV Future Work (cont.)

C. METALLOGRAPHY AND ELECTRON MICROSCOPY

Photomicrographs of selected cracked specimens are being prepared and studied. In addition, the cracking process is being studied by means of the electron microscope, utilizing fracture replicas. Photographs of these replicas have been made. These results are being evaluated, while further studies are in progress. The intention is to attempt to define the mode of failure and, if possible, associate the failure process with microstructural characteristics of the materials. Evaluation of some of these results will be presented in the next quarterly report.

TABLE 1
MASTER SCHEDULE, FIFTH-YEAR PROGRAM

| Alloy | 0.2% Yield Strength* | Possible Heat Treatment | Test Method | Code | Distilled Water (-01) | % NaCl (-02) | Sodium Dichromate Solution (-04) | Soluble Oil (-05) | High Humidity (-06) | Trichloroethylene (-07) | Cosmoline (-08) | Solid Propellant (-09) | Air Exposure (-10) | Sea Coast Exposure (-11) |
|---------------------------|-----------------------|---|--------------|-------|-----------------------|--------------|----------------------------------|-------------------|---------------------|-------------------------|-----------------|------------------------|--------------------|--------------------------|
| CAL-4V Titanium | 138,000 | As received, annealed | Bent Beam | G-1-B | 3** | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| | | | U-Bend | G-1-U | - | - | - | - | 3 | 3 | - | - | 2 | 3 |
| | | | Center Notch | G-1-C | 2 | 2 | 2 | 2 | - | - | - | - | - | - |
| | 143,000 | 1650°F WQ and 900°F age | Bent Beam | G-2-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| 20-Nickel Maraging Steel | 110,000 (anticipated) | Welded, 900°F age | U-Bend | G-2-U | - | - | - | - | 3 | 3 | - | - | 2 | - |
| | | | Center Notch | G-2-C | 2 | 2 | 2 | 2 | - | - | - | - | - | - |
| | | Solution anneal -100°F, 850°F age | Bent Beam | G-W-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| | | | U-Bend | G-W-U | - | - | - | - | 3 | 3 | - | - | - | 3 |
| 20-Nickel Maraging Steel | 291,000 | Solution anneal -100°F, 850°F age | Bent Beam | H-1-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| | | | U-Bend | H-1-U | - | - | - | - | 3 | 3 | - | - | 2 | 3 |
| | 295,000 (anticipated) | 50% CW*** 850°F age | Center Notch | H-1-C | 2 | 2 | 2 | 2 | - | - | - | - | - | - |
| | | | Bent Beam | H-2-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| 18-NiMoCo Maraging Steel | 298,000 | 7% CW 850°F age | U-Bend | H-2-U | - | - | - | - | 3 | 3 | - | - | 2 | - |
| | | | Center Notch | H-2-C | 2 | 2 | 2 | 2 | - | - | - | - | - | - |
| | 250,000 (anticipated) | Welded | Bent Beam | H-3-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| | | | U-Bend | H-3-U | - | - | - | - | 3 | 3 | - | - | 2 | - |
| 18-NiMoCo Maraging Steel | 283,000 | 0% CW 0.6% Ti Aged at 900°F | Center Notch | H-3-C | 2 | 2 | 2 | 2 | - | - | - | - | - | - |
| | | | Bent Beam | H-W-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| | 300,000 (anticipated) | 0.6% Ti Aged at 900°F | U-Bend | H-W-U | - | - | - | - | 3 | 3 | - | - | - | 3 |
| | | | Center Notch | I-1-C | 2 | 2 | 2 | 2 | - | - | - | - | - | - |
| 9Ni-4Co Vacuum-Cast Alloy | 324,000 | 50% CW 0.3/0.6% Ti _{0.2} Aged at 900°F | Bent Beam | I-2-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| | | | U-Bend | I-2-U | - | - | - | - | 3 | 3 | - | - | 2 | - |
| | No data on hand | 0.6% Ti Aged at 900°F | Center Notch | I-2-C | 2 | 2 | 2 | 2 | - | - | - | - | - | - |
| | | | Bent Beam | I-3-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| 9Ni-4Co Vacuum-Cast Alloy | No data on hand | 0.6% Ti Aged at 900°F | U-Bend | I-3-U | - | - | - | - | 3 | 3 | - | - | 2 | - |
| | | | Center Notch | I-3-C | 2 | 2 | 2 | 2 | - | - | - | - | - | - |
| | No data on hand | Welded | Bent Beam | I-W-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| | | | U-Bend | I-W-U | - | - | - | - | 3 | 3 | - | - | - | 3 |
| 9Ni-4Co Vacuum-Cast Alloy | No data on hand | Aged*** 0.25%/0.30% C | Bent Beam | J-1-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| | | | U-Bend | J-1-U | - | - | - | - | 3 | 3 | - | - | - | 3 |
| | No data on hand | Aged*** 0.40/0.45% C | Center Notch | J-1-C | 2 | 2 | 2 | 2 | - | - | - | - | 2 | - |
| | | | Bent Beam | J-2-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| 9Ni-4Co Vacuum-Cast Alloy | No data on hand | Aged*** 0.40/0.45% C | U-Bend | J-2-U | - | - | - | - | 3 | 3 | - | - | - | 3 |
| | | | Center Notch | J-2-C | 2 | 2 | 2 | 2 | - | - | - | - | - | - |
| | No data on hand | Aged*** 0.40/0.45% C | Bent Beam | J-3-B | 3 | 3 | 3 | 3 | 3 | - | 3 | 3 | 3 | 3 |
| | | | U-Bend | J-3-U | - | - | - | - | 3 | 3 | - | - | - | 3 |

*Based on Aerojet tests.

**Number refers to number of tests.

***Indicates material not yet received.

Table 1

TABLE 2
CHEMICAL ANALYSIS AND MECHANICAL PROPERTIES OF MARAGING STEELS

| Mill-Certified Analysis (Percent Composition) | | | | | | | | | | | |
|---|-------|-------|-------|-------|-------|------|------|-------|------|-------|-------|
| C | Mn | P | S | Si | Ni | Co | Mo | Al | Cr | Ti | B |
| 0.009 | 0.09 | 0.002 | 0.005 | 0.06 | 20.31 | - | - | 0.29 | 0.59 | 0.002 | 0.004 |
| 0.012 | 0.01 | 0.003 | 0.005 | 0.01 | 13.59 | 3.90 | 4.32 | 0.29 | - | 0.02 | 0.002 |
| 0.013 | 0.002 | 0.006 | 0.004 | 0.024 | 13.39 | 3.10 | 4.35 | 0.089 | - | 0.40 | - |
| 0.023 | 0.002 | 0.004 | 0.003 | 0.009 | 13.31 | 3.46 | 4.32 | 0.089 | - | 0.52 | - |
| 0.020 | 0.002 | 0.006 | 0.005 | 0.014 | 13.60 | 3.05 | 4.30 | 0.078 | - | 1.00 | - |

| Mechanical Properties (Aerocet Tests) | | | | | | | |
|---------------------------------------|---------------------|------------------|-------------------------------------|---------------------------|-------------------------------|--------------------------------|-----------------------|
| Percent Cold Reduction | Aging Treatment | Table 1 Code No. | 0.2% Offset Y.S. (psi) (Transverse) | U.T.S. (psi) (Transverse) | Percent Elongation (in 2 in.) | Notched Tensile Strength (psi) | Hardness (Rockwell C) |
| | | | | | | | |
| 0 | None | H-1 | 128,500 | 170,700 | 7.5 | - | 34 |
| 0 | -100°F + 350°F + nr | H-1 | 291,500 | 302,200 | 5 | 55,200 | 34 |
| 50 | None | H-2 | 135,100** | 197,300** | 40** | - | 32** |
| 50 | 350°F 4 hr | H-2 | 319,200** | 322,700** | 5** | - | 35** |
| 75 | None | H-3 | 205,700 | 220,900 | - | - | 35 |
| 75 | 350°F 4 hr | H-3 | 298,500 | 308,500 | 2.5 | 31,500 | 35 |
| 0*** | None | I-1 | 132,000 | 153,300 | 22 | - | 30.5 |
| 0*** | 900°F 3 hr | I-1 | 283,000 | 294,000 | 38 | - | 33.5 |
| 50 | None | I-3 | 157,700 | 189,000 | 31 | - | 32.5 |
| 50 | 900°F 3 hr | I-3 | 323,500 | 325,403 | 28 | - | 35 |
| 50 | None | - | 109,500 | 196,900 | 0.5 | - | 33.5 |
| 50 | 900°F 3 hr | - | 278,000 | 290,700 | 2 | - | 35 |
| 0 | None | - | 105,500 | 150,500 | 10 | - | 30.5 |
| 0 | 900°F 3 hr | - | 255,400 | 265,900 | 5 | - | 32 |
| 50 | None | - | 175,500 | 199,500 | 4.5 | - | 36 |
| 50 | 900°F 3 hr | - | 331,000 | 332,500 | 1.5 | - | 35 |
| 0 | None | - | 128,500 | 174,700 | 5.5 | - | 35 |
| 0 | 900°F 3 hr | - | 323,500 | 330,000 | 2.5 | - | 35 |
| 50 | None | - | 192,200 | 217,000 | 2.5 | - | 34 |
| 50 | 900°F 3 hr | - | 354,403 | 354,900 | 1 | - | 35 |

* Tensile tests of fatigue-cracked specimens shown in Figure 3.

** Mill report.

*** Re-annealed, cold-worked material.

Table 2

TABLE 3CHEMICAL ANALYSIS AND MECHANICAL PROPERTIES
OF 6Al-4V TITANIUM

| | Chemical Analysis (% Composition)* | | | | | | | | |
|------------------|------------------------------------|-----------|----------|----------------------|----------------------|----------------------|-----------|-----------|--------------|
| | <u>C</u> | <u>Al</u> | <u>V</u> | <u>O₂</u> | <u>N₂</u> | <u>H₂</u> | <u>Ti</u> | <u>Fe</u> | <u>Other</u> |
| Aerojet Analysis | 0.03 | 6.1 | 4.1 | 0.083 | 0.014 | 80 ppm | Bal | 0.16 | 0.18 |

| | Mechanical Properties (Transverse) | | | | |
|-----------------------------------|---|--|---------------------------------|---|------|
| | <u>Yield Strength</u> <u>(0.2% offset)</u> <u>(psi)</u> | <u>Ultimate</u> <u>Strength</u> <u>(psi)</u> | <u>Elongation</u> <u>(%)</u> | <u>Hardness</u> <u>(R_c)</u> | |
| Annealed | | | | | |
| Mill report | | 131,900 | 141,400 | 12 | 33.5 |
| Aerojet test | | 138,000 | 143,800 | 14 | 34 |
| Notched tensile strength** | -- | 128,500 | - | - | - |
| 1675°F 1 hr, W.Q. Aged 900°F 8 hr | | | | | |
| Aerojet test | | 162,700 | 176,800 | 7 | 38.5 |
| Notched tensile strength | -- | 132,000 | - | - | - |

*Titanium Metals Corporation HT 4141.

**Using as-fatigue-cracked sample of Figure 3.

TABLE 4

STRESS CORROSION OF 6Al-4V TITANIUM
IN VARIOUS ENVIRONMENTS

| Environment | Condition G-1 [*] Annealed (as received) | | | Condition G-2 1675 ^o F 1 hr, W.Q., 900 ^o F 8 hr | | |
|--------------------------|---|---------------|---------------|---|---------------|---------------|
| | Failed/Tested | Failure Times | | Failed/Tested | Failure Times | |
| | | Mean (hr) | Range (hr) | | Mean (hr) | Range (hr) |
| Bent Beam Tests | | | | | | |
| Distilled water | 0/3** | - | NF780*** | 0/3 | - | NF780 |
| Tap water | 0/3 | - | ↓ | 0/3 | - | ↓ |
| 3% NaCl sol. | 0/3 | - | | 0/3 | - | |
| 0.25% Sodium dichromate | 0/3 | - | | 0/3 | - | |
| Soluble oil sol. | 0/2 | - | | 0/3 | - | |
| Cosmoline | 0/3 | - | ↓ | 0/3 | - | ↓ |
| High-humidity atmosphere | 0/3 | - | 0/3 | - | | |
| Air | 0/3**** | - | NF780 | 0/3 | - | NF780 |
| Solid propellant | 0/0 | - | - | 0/0 | - | - |
| Sea-coast exposure | 0/0 | - | - | 0/0 | - | - |
| U-Bend Tests | | | | | | |
| High-humidity atmosphere | 0/0 | - | - | 0/3 | - | NF310 |
| Trichloroethylene | 0/0 | - | - | 0/0 | - | - |
| Sea-coast exposure | 0/0 | - | - | 0/0 | - | - |
| Center-Notch Tests | | | | | | |
| Distilled water | 0/2 | - | NF100 | 0/2 | - | NF100 |
| 3% NaCl sol. | 0/2 | - | ↓ | 0/2 | - | ↓ |
| 0.25% Sodium dichromate | 0/2 | - | ↓ | 0/2 | - | ↓ |
| Soluble oil sol. (4%) | 0/1 | - | NF100 | 0/1 | - | NF100 |

* Refers to code letter in Master Schedule, Table 1.

** Indicates no failures of three samples exposed.

*** Indicates no failures in 780 hours exposure.

**** Indicates testing not started.

TABLE 5

STRESS CORROSION OF 20%-NICKEL
MARAGING STEEL IN VARIOUS ENVIRONMENTS

| Environment | Condition H-1 [*] Solution Anneal -100°F, 850°F 4 hr | | | Condition H-3 75% Cold Work 850°F 4 hr | | |
|--------------------------|---|---------------|----------------------|--|---------------|---------------|
| | Failed/Tested | Failure Times | | Failed/Tested | Failure Times | |
| | | Mean (hr) | Range (hr) | | Mean (hr) | Range (hr) |
| Bent Beam Tests | | | | | | |
| Distilled water | 3/3 ^{**} | 11 | 10.2-18 | 0/3 | - | NF600 |
| Tap water | 0/3 | - | NF600 ^{***} | 0/3 | - | ↓ NF600 |
| 3% NaCl sol. | 3/3 | 7.3 | 6-8.5 | 0/3 | - | |
| 0.25% Sodium dichromate | 1/3 | 1 | 1-NF600 | 0/3 | - | |
| Soluble oil sol. | 0/3 | - | NF600 | 0/3 | - | |
| Cosmoline | 0/3 | - | NF600 | 0/3 | - | |
| High-humidity atmosphere | 3/3 | 100 | 22-174 | 0/3 | - | NF600 |
| Air | 0/3 | - | NF600 | 0/3 | - | |
| Solid propellant | 0/0 ^{****} | - | - | 0/0 | - | - |
| Sea-coast exposure | 0/0 | - | - | 0/0 | - | - |
| U-Bend Tests | | | | | | |
| High-humidity atmosphere | 0/0 | - | - | 0/0 | - | - |
| Trichlorethylene | 0/0 | - | - | 0/0 | - | - |
| Sea-coast exposure | 0/0 | - | - | 0/0 | - | - |
| Center-Notch Tests | | | | | | |
| Distilled water | 3/3 | 5.1 | 4.3-6.6 | 0/0 | - | - |
| 3% NaCl sol. | 2/2 | 7.2 | 6.6-7.8 | 0/0 | - | - |
| 0.25% Sodium dichromate | 0/2 | - | NF60 | 0/0 | - | - |
| Soluble oil sol. (4%) | 0/1 | - | NF60 | 0/0 | - | - |
| Air | 0/0 | - | - | 0/0 | - | - |

^{*} Refers to code letter of Table 1, Master Schedule.

^{**} Indicates three failures of three samples tested.

^{***} Indicates no failures in 600 hours of testing.

^{****} Indicates testing not started.

TABLE 6STRESS CORROSION OF 18%-NICKEL
MARAGING STEEL IN VARIOUS ENVIRONMENTS

| Environment | Condition I-1* 50% C.W., Annealed 1500°F 1 hr, Aged 900°F 3 hr | | | Condition I-3 50% C.W. Aged 900°F 3 hr | | |
|--------------------------|--|-----------|------------|--|-----------|---------------|
| | Failure Times | | | Failure Times | | |
| | Failed/Tested | Mean (hr) | Range (hr) | Failed/Tested | Mean (hr) | Range (hr) |
| Bent Beam Tests | | | | | | |
| Distilled water | 0/0** | - | - | 1/2*** | 440 | 440-NF445**** |
| Tap water | 0/0 | - | - | 0/2 | - | NF445 |
| 3% NaCl | 0/0 | - | - | 0/2 | - | NF445 |
| 0.25% Sodium dichromate | 0/0 | - | - | 0/2 | - | NF445 |
| Soluble oil sol. | 0/0 | - | - | 0/2 | - | NF445 |
| High-humidity atmosphere | 0/0 | - | - | 2/2 | 260 | 245-290 |
| Cosmoline | 0/0 | - | - | 0/2 | - | NF445 |
| Solid propellant | 0/0 | - | - | 0/0 | - | - |
| Air | 0/0 | - | - | 0/0 | - | - |
| Sea-coast exposure | 0/0 | - | - | 0/0 | - | - |
| U-Bend Tests | | | | | | |
| High-humidity | 0/0 | - | - | 0/0 | - | - |
| Trichloroethylene | 0/0 | - | - | 0/0 | - | - |
| Sea-coast exposure | 0/0 | - | - | 0/0 | - | - |
| Center-Notch Tests | | | | | | |
| Distilled water | 0/0 | - | - | 0/0 | - | - |
| 3% NaCl sol. | 0/0 | - | - | 0/0 | - | - |
| 0.25% Sodium dichromate | 0/0 | - | - | 0/0 | - | - |
| Soluble oil (4% sdn) | 0/0 | - | - | 0/0 | - | - |
| Air | 0/0 | - | - | 0/0 | - | - |

* Refers to code letter of Table 1, Master Schedule.

** Indicates tests not started.

*** Indicates one failure of two samples tested.

**** Indicates 445 hours without failure.

TABLE 7EVALUATION OF PROTECTIVE COATINGS ON H-11 STEEL
(FOR PREVENTING STRESS-CORROSION CRACKING)

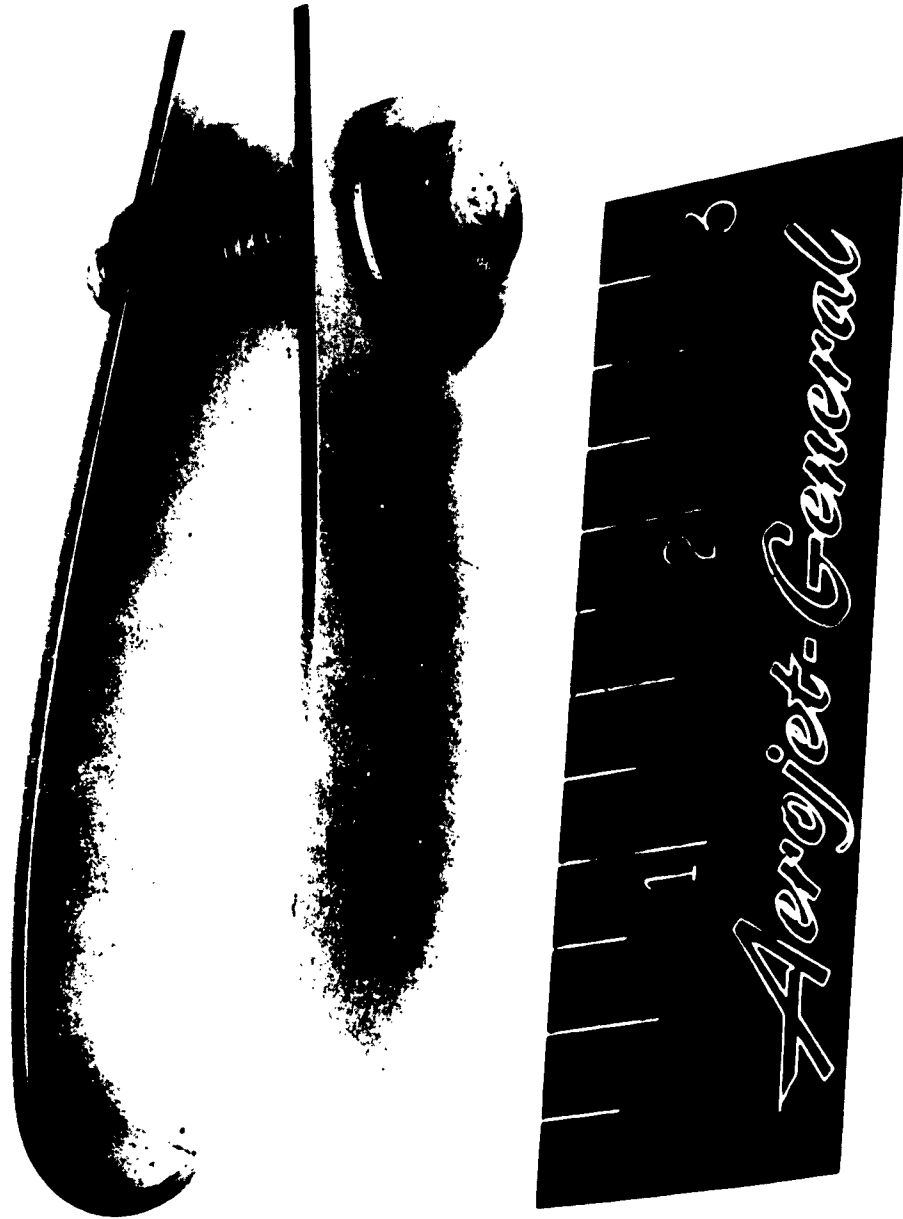
| | <u>140°F High Humidity Atmosphere</u> | | | <u>3% NaCl Solution</u> | | |
|-----------------------|---------------------------------------|----------------------------|-----------------------------|-------------------------|----------------------------|-----------------------------|
| | <u>Failed/Tested</u> | <u>Failure Times</u> | | <u>Failed/Tested</u> | <u>Failure Times</u> | |
| | | <u>Mean</u> <u>(hr)</u> | <u>Range</u> <u>(hr)</u> | | <u>Mean</u> <u>(hr)</u> | <u>Range</u> <u>(hr)</u> |
| H-11 Steel (uncoated) | 2/2* | 64 | 48-70 | 2/2 | 1.7 | 0.8-2.5 |
| Epoxy 463-4-8 | 1/3 | 289 | 289-NF570** | 3/3 | 550 | 525-578 |
| Epoxy 463-1-5 | 1/3 | 400 | 400-NF570 | 0/3 | - | NF700 |
| Zinc silicate | 1/2 | 170 | 170-NF440 | 2/2 | 1.2 | 0.8-1.6 |
| 80% Aluminum-epoxy | 2/2 | 30 | 16-45 | 2/2 | 100 | 100 |
| 70% Titanium-epoxy | 1/2 | 150 | 150-NF240 | 2/2 | 150 | 140-160 |

* Indicates two failures out of two samples exposed.

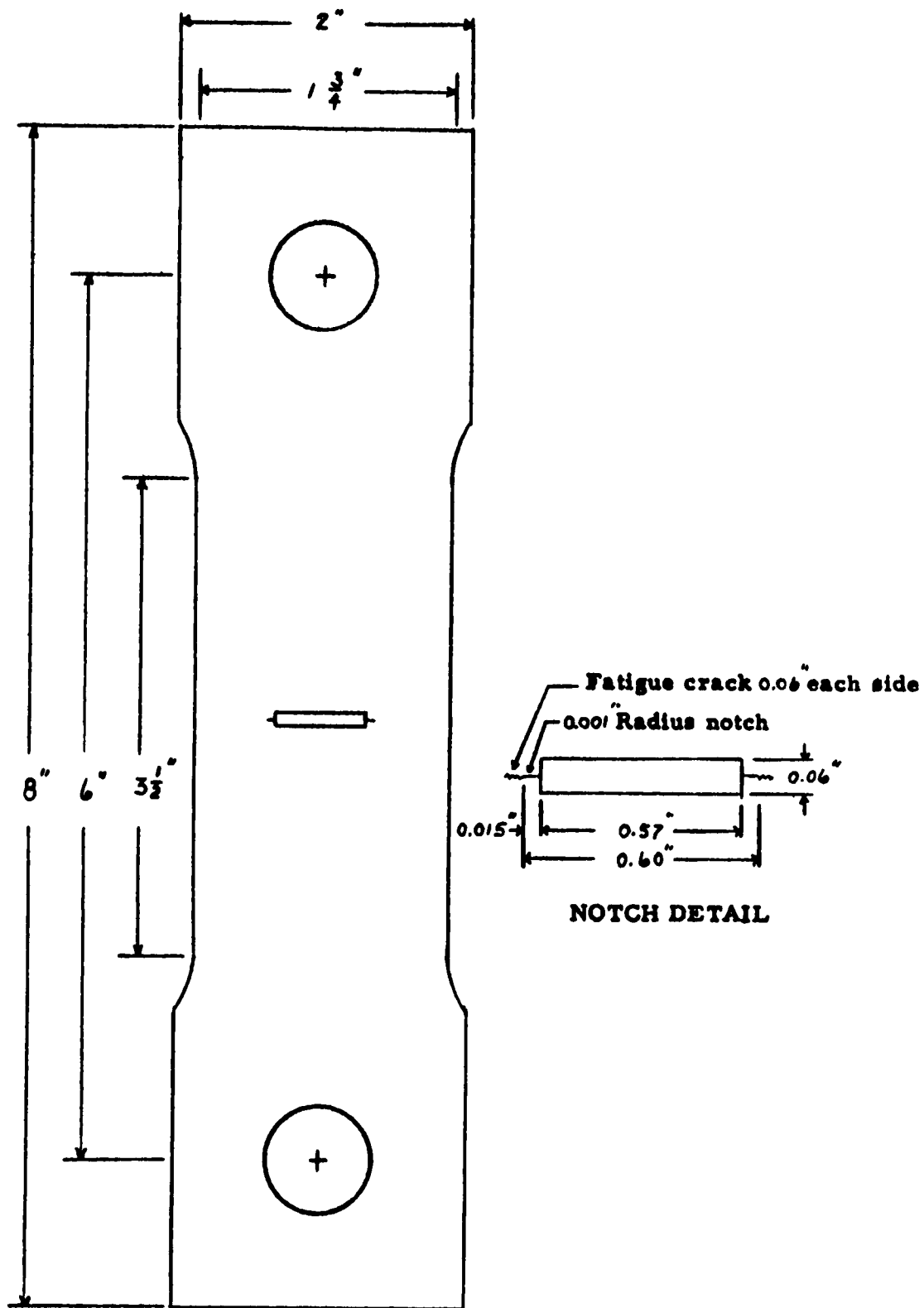
** Indicates no failure in 570 hours.



Insulated Phelps Bent-Beam Specimens

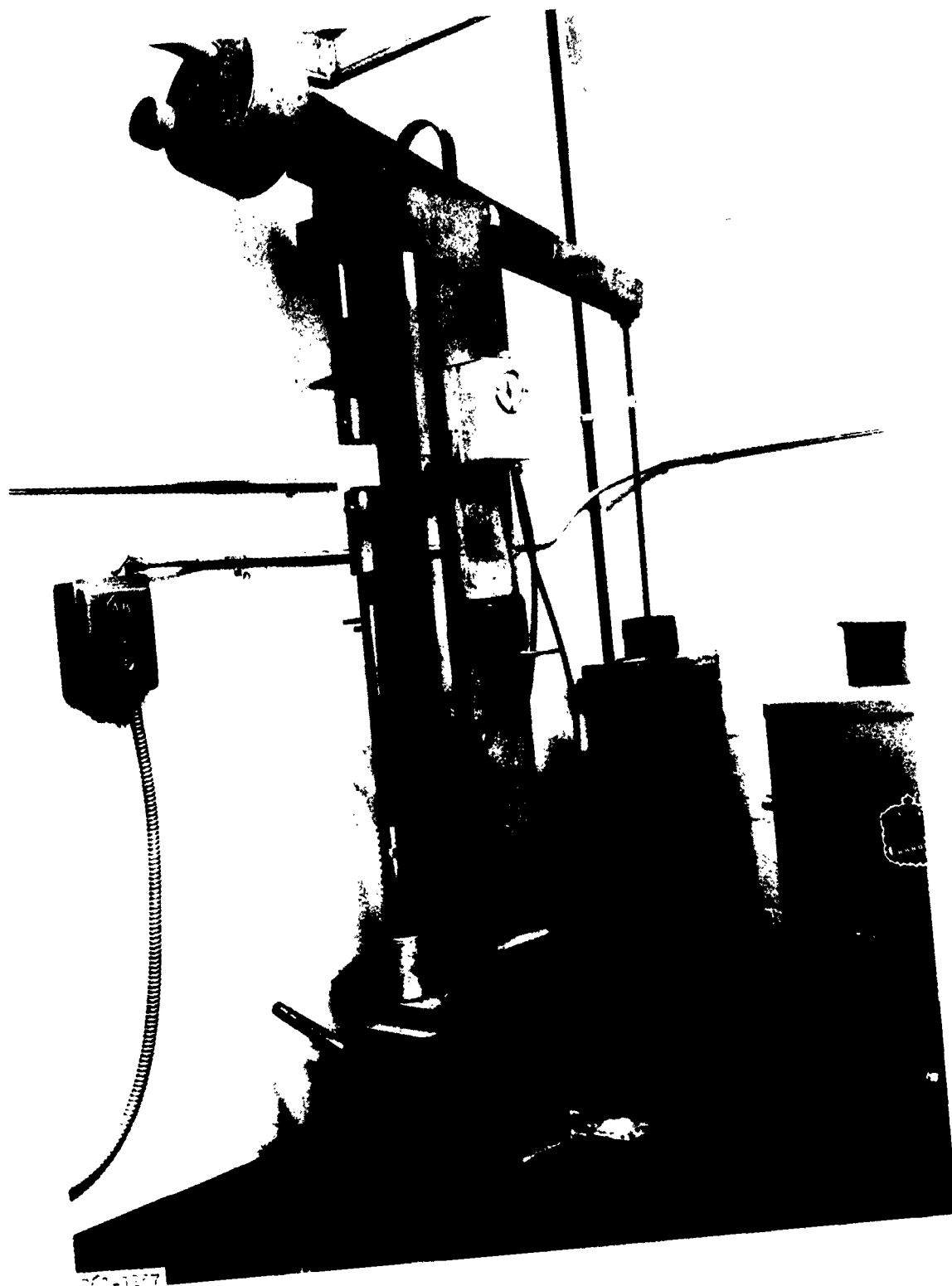


U-Bend Test Specimen



ELOX-NOTCHED SPECIMEN
FOR CRACK PROPAGATION STUDY

Figure 3



Stress-Corrosion Test Setup for Center-Notched Specimens

Figure 1

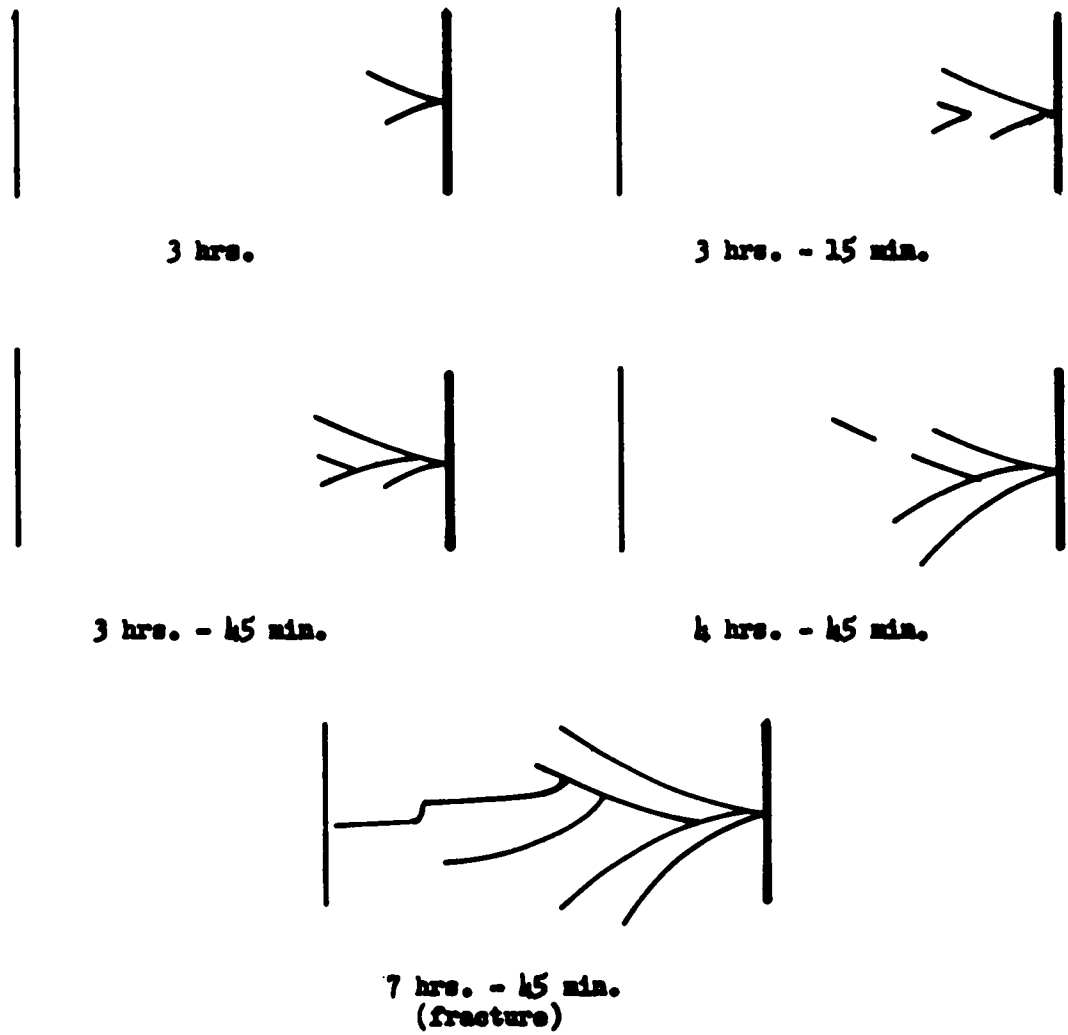


Crack Pattern on Surface of Beam Sample after 10 hours in Aerated Distilled Water. (5X)



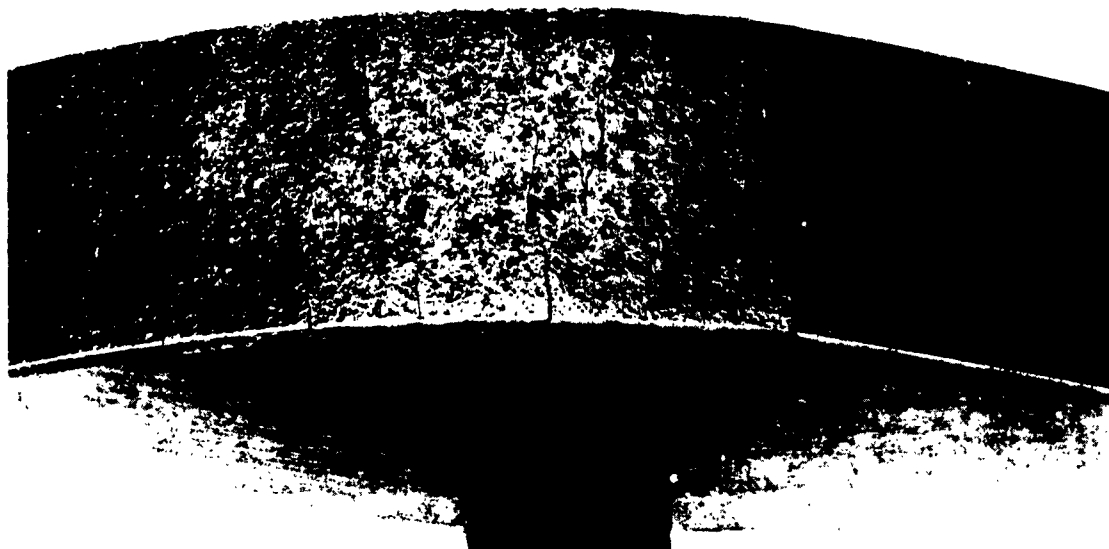
Vertical View of Cracking in Above Sample Showing Intergranular Cracks. Etchant is Diluted Marbles Reagent (1000X)

Stress-Corrosion Crack Pattern on 20%-Nickel Maraging Steel



Crack Propagation Study on 20%-Nickel Maraging Steel in Salt Water

Figure 6

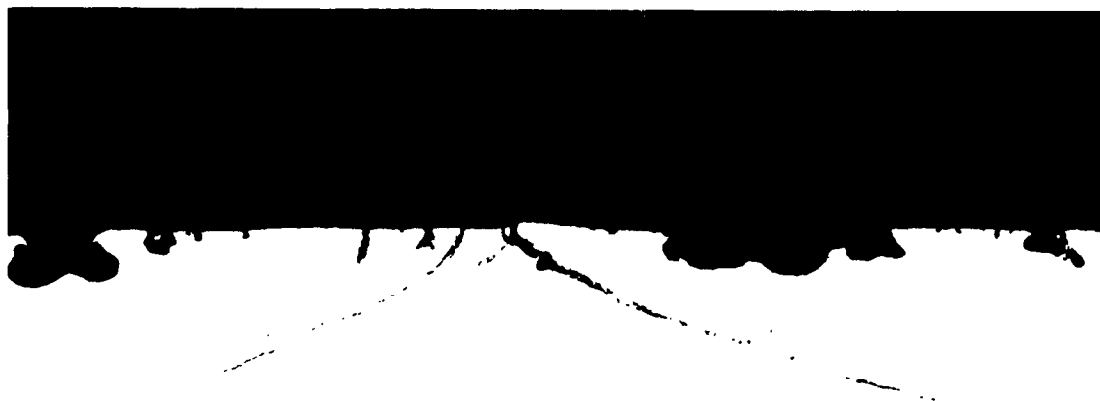


Surface of Cold-Worked-and-Aged 18%-Nickel Maraging Steel After 10-Days at 140°F in High Humidity Stress Corrosion Test. Surface has been Wire-Brushed. (Approx. 2X)



Cross-Section of Above Sample Showing Possible Cracking Along Slip Planes. (10X)

Stress-Corrosion Crack Pattern on 20%-Nickel Maraging Steel



View of Surface in Lightly Cracked Area Showing Pitting Attack (100X)



General Structure in Interior of Highly Cracked Area.
Etchant - 10% Ammonium Persulphate-electrolytic. (500X)

Photomicrographs of Stress-Corrosion Cracking in 18%-Nickel Maraging Steel



AEROJET-GENERAL CORPORATION